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# TECHNICAL NOTE

## D-76

DUCTILE CERAMICS

II - INTRODUCTORY STUDY OF DUCTILITY IN POLYCRYSTALLINE

SODIUM CHLORIDE AND MAGNESIUM OXIDE

By Charles E. May, Hubert H. Grimes, and Robert A. Lad

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SUMMARY

    Methods for producing ductile polycrystalline ceramic-type materials were investigated. Various types of treatments were used for sodium chloride; these can be divided into three categories: surface treatments of the crystallites, surface treatments of the compacted specimens, and bulk treatments. The most effective method for producing ductility was to deposit a small quantity of silver on the individual crystallites by vapor deposition. The silver apparently acts as a specific surface-active agent. A treatment using stearic acid was also successful in producing ductility, but it was not as effective as that using silver.

    Thus far, no ductility has been produced in polycrystalline magnesium oxide, since the required sintering causes loss of the desired metallic deposits.

INTRODUCTION

    Ceramic materials have been considered for use in high-temperature structural applications in air and space craft. The greatest disadvantage of ceramics is the extreme brittleness which in the past was believed to be an intrinsic property of these ionic substances. Recently, it has been shown that single crystals of ionic substances such as magnesium oxide, lithium fluoride, and sodium chloride can be made to exhibit considerable ductility in bending tests at room temperature (refs. 1 to 4). The condition of the crystal surfaces has an extremely important bearing on the ductility of a sample. It was reasoned that polycrystalline ceramics might also be made to exhibit ductility if the specimen surfaces were properly treated. Since more is known about the ductility of sodium chloride than other single crystals, sodium chloride was the first polycrystalline material investigated at the Lewis Research Center. This report describes two treatments which were successful (a

silver deposit and stearic acid coating) and many which were unsuccessful in producing ductility.

Difficulties arise in attempts to apply some of the treatments used on sodium chloride to a true ceramic material such as magnesium oxide. Sodium chloride has the desirable property of sintering at room temperature during pressing. This is not true of magnesium oxide, for which sintering at high temperature is required (ref. 5). Under these temperature conditions surface deposits such as stearic acid and silver should be, respectively, thermally decomposed and volatilized. However, a coating might still be effective in producing ductility if it induces the effect during the pressing process and does not have to be present during the actual bending test. With this in mind, a few introductory experiments were performed on magnesium oxide.

The work with sodium chloride is described in the first two sections; these are followed by a description of the work on magnesium oxide.

EXPERIMENTAL PROCEDURE FOR POLYCRYSTALLINE SODIUM CHLORIDE

The polycrystalline specimens were formed by compacting powdered sodium chloride prepared from Harshaw melt-grown single crystals. The salt was ball-milled to pass a 325-mesh screen. The compact was made by placing about 10 grams of the powder in 5/8-inch intravenous tubing (which in turn was placed in 3/4-in. intravenous tubing) and applying hydraulic pressure (50,000 lb/sq in.). Specimens were cut and ground to size (0.125 by 0.25 by 1.6 in.).

The specimens were then tested in flexure using the four-point-loading jig shown in figure 1. The tests were run at a constant deflection rate of 0.01 inch per minute in an Instron tensile testing machine which recorded the load as a function of deflection.

The treatments used can be divided into three major categories as shown in table I: surface treatment of the crystallites before compacting into specimens, surface treatment of the compacted specimens, and bulk treatment (either of the compacted or uncompacted material). The reason for using many of the treatments was that similar treatments proved to be successful for increasing the ductility of single-crystal sodium chloride (ref. 1). Other treatments were used in order to obtain an understanding of the mechanism for the successful treatments.

The first type of surface treatment used on the crystallites before compacting was a metal vapor deposition. The metal was deposited on the sodium chloride powder in the apparatus shown schematically in figure 2. The container holding the sodium chloride crystallites was inclined at

an angle about 45° from the vertical and was rotated about this 45° axis. The rotation of the container, which was equipped with fins and a hammer mechanism that struck the container every quarter rotation, tumbled the sodium chloride so that new crystal surfaces were constantly exposed. Fine wire of the metal to be deposited (e.g., silver, gold, copper) was wound on the spiral of heavier (20 mil) tungsten wire, which was then heated electrically in vacuum (0.1 to 0.5 microns Hg). Metal thus evaporated was condensed on the surfaces of the sodium chloride. During the period of vaporization (about 4 hr) the surfaces of each crystal were exposed to vaporization of metal on the average of 20 times. The sample was then compacted in the manner described previously.

The second type of surface treatment of the crystallites before compacting included solution treatments which either removed the surfaces or deposited a foreign material on the surfaces. This was accomplished (unless otherwise specified in table I) by placing the uncompacted crystallites in the solution, stirring for 3 to 5 minutes, and then removing the liquid. The last trace of the liquid present in the solid material was allowed to evaporate. The material was then pressed in the usual manner.

The third type of treatment was the exclusion of air during ballmilling, screening, and pressing of the crystallites. The operations were carried out in a helium atmosphere.

Similar types of treatments were performed on the compacted specimens. Silver was vapor-deposited onto the compression and tension sides of the specimen. For the liquid treatments of the compacted samples, the specimens were merely submerged in the liquid several seconds and then removed; they were tested while still slightly wet.

In the category of bulk treatments, two were tried. For the first treatment, the crystallites were X-irradiated for various amounts of time while being rotated in a light-tight container filled with a helium atmosphere. The X-ray unit was run at 50 kilovolts and 30 milliamperes using a tungsten target with the sample 8 inches from the target. The irradiated powers and the subsequently compacted specimens were stored in the dark before testing to avoid bleaching. The second bulk treatment was an annealing of the compacted specimens in vacuum for 15 hours at 1000° F.

Variations of the treatments listed above are noted in table I. In general, duplicate samples were tested. Specimens of particular interest were analyzed chemically for the substance which was added to the surfaces during the preparation of the samples. The thicknesses of the surface deposits were then calculated from the analyses and from the grain size of the crystallites, assuming uniform plating.

Two assumptions have been made in these calculations: the grain
size of powder passing a 325-mesh screen was taken to be 40 microns, and
the grains were assumed to be perfect spheres. Because of these assump-
tions in the calculations, the average thicknesses listed in table I may
be too large by a factor of 10. Furthermore, vapor deposition of thin
deposits of metal (of the order of 50 Å and less) do not produce a uni-
form coating; some parts of the surface do not become plated, while
others obviously have deposits greater than the average thickness.
Therefore, the thicknesses reported in table I are nominal and are given
for the purpose of comparison.

## DISCUSSION OF RESULTS FOR SODIUM CHLORIDE

Typical curves for untreated specimens are shown in figure 3, where
load is plotted against deflection. Each of these curves exhibits an
initial nonlinear "foot" followed by a linear region which terminates at
the fracture point. A calculated Hooke's law curve is plotted for com-
parison (see the appendix for details of computation); the published
value, $4.0 \times 10^{11}$ dynes per square centimeter, was used for Young's modu-
lus (ref. 6). There is fair agreement between the linear slopes of the
experimental curves for polycrystals and the slope of the Hooke's law
curve. The initial "foot" has been interpreted to be due to anelastic
behavior of the specimen (ref. 1). The foot length for an untreated
single crystal is similar to that for a polycrystal (fig. 3). However,
in the region of the curve following the anelastic region, behavior of a
polycrystal differs from that of a single crystal. Single crystals of
sodium chloride always exhibit ductility (ref. 1), while untreated poly-
crystalline samples have never shown ductility.

The majority of the treatments described in this report resulted in
no ductility in bending tests (table I). Ductility in this report is de-
fined as deviation from linear (Hooke's law) behavior at reasonably large
loads. Only two types of treatments were successful in producing signifi-
cant ductility: vapor deposition of silver onto the surfaces of the
crystallites before compacting and solution deposition of stearic acid
onto the surfaces of the crystallites before compacting.

### Effect of Silver Deposits

The first results to be discussed are those of specimens with silver
deposited on the uncompacted crystallites of sodium chloride. This
treatment was tried because a silver deposit on a single crystal produced
a definite increase in ductility. In the polycrystalline material it
produced a similar effect even at low loads, as can be seen in figure 4.
The curves shown are the results for samples containing various amounts
of silver on the crystalline surfaces. Since the values listed on the

graph are the nominal thicknesses of deposits around the grains, the average thickness between the adjacent grains is twice this value. A 2 Å deposit was not sufficient to produce ductility; a 5 Å deposit did produce ductility; a 7 Å deposit produced even more, as indicated by a greater deviation from linearity. A 12 Å deposit was the thickest layer tried; these samples exhibited about four times as much deflection as an untreated specimen. The area under each curve is related to the energy which is absorbed in bending the ceramic material. The area under the 12 Å curve is over three times the area under the curve for an untreated sample. However, the rupture of the treated specimens occurred at lower loads than for an untreated one, whereas for single crystals treatments that increased ductility also increased strength.

The ductile portion of these curves is preceded by a foot similar to the one present for the curve of the untreated specimen. There is no slope on the load-deflection curve of any of the ductile specimens which is in agreement with the correct modulus for sodium chloride; this is similar to the results for untreated single crystals of sodium chloride (ref. 1).

It was believed that the ductility produced by silver might depend upon the exposure to air of the crystallite surfaces immediately before the silver was deposited. The specimens with the 12 Å deposit of silver on the crystallites were samples fabricated to determine this effect. The original single crystal stock from which these samples were made was ground under chloroform, the chloroform was evaporated in vacuum, and the crystallites (unscreened) were not exposed to air until after the silver was vapor-deposited upon them. Such crystallites might have their as-cleaved surfaces (with no air adsorbed) preserved by the coating of silver. However, from the fact that the specimens with a 12 Å deposit fit the general progression of ductility with thickness, it can be inferred that exclusion of air did not have a large effect on ductility.

The samples for which the silver was deposited on the surfaces of the compacted specimens showed no ductility. This result indicates that the silver is not working in a macroscopic mechanical manner so as to prevent the origination of cracks on the tension side of the test specimen. The effectiveness of the silver is associated with the individual crystallites.

Since silver chloride was found in addition to metallic silver by chemical analysis of samples with the silver deposit on the individual crystallites, the possibility existed that the silver chloride might have been the active agent for producing ductility. However, specimens for which silver chloride was vaporized onto the individual crystals showed no ductility; this indicates that silver chloride is not responsible for the ductility.

No ductility was found for specimens for which metals similar to silver (copper and gold) were vapor-plated onto the individual crystallites. The fact that copper and gold are not effective indicates that silver is a very specific agent. Such specificity is shown in epitaxy experiments (ref. 7) which indicate that sodium chloride orients more readily on silver than on either gold or copper even though the differences between the lattice parameters of silver, gold, and copper are quite small.

Although metals similar to silver were unsuccessful in producing ductility, there is the possibility that other metals might produce the desired effect. The only two others tried were magnesium and chromium; they were unsuccessful.

It was first believed that, if the silver was a surface-active material, a composite deposit, in which the silver was plated first and the copper second, would produce a material which would also be ductile. However, a composite deposit with the plating in the reverse order should not produce a ductile material. Both types of composites were tried and found to be unsuccessful in producing ductility.

The most useful way to interpret the effectiveness of silver in producing ductility in sodium chloride is to consider the silver to be a very specific surface-active agent. The dependence of ductility on the thickness of the deposit is interpreted as being due to incomplete coverage of the crystallites with small thicknesses of silver. The results with specimens having composite vapor platings may then be interpreted to be due to contamination of the crystallite surfaces with copper, since such contamination could occur regardless of the order of application of the vapor plates.

## Effect of Stearic Acid

Specimens for which the crystallites were coated with stearic acid were found to exhibit ductility, as shown in figure 5. The curves shown in this figure are much like those for the specimens with silver deposits; each has a small foot, no portion of the curve has the slope corresponding to the correct modulus, and the maximum strain is several times that for an untreated specimen. The load at rupture is lowered and the area under the curve is several times that for an untreated specimen. Also, the ductility depends on the thickness of the layer, since a 0.2-micron layer produces very little ductility compared with a 0.5-micron layer. However, the thickness of stearic acid required for a reasonable amount of ductility exceeds the thickness of silver required for similar ductility by a factor of about 500. Because of the thick layer of stearic acid required, the ductility produced can be interpreted as being due to the stearic acid acting as a lubricant in the grain boundaries. The thickness

of silver required for similar ductility is so small that the ductility produced by silver is not easily accounted for by such a hypothesis; the ineffectiveness of gold and copper also make such a hypothesis unsatisfactory for the case of silver.

In the case of a stearic acid, two slightly different methods of application were used, as is noted in table I. Any variation of the load-deflection curves between the two methods of application is probably due to the relative effectiveness in dispersing the stearic acid throughout the sodium chloride powder. Little or no ductility was observed in the compacted and cut specimen of sodium chloride to which the stearic acid was applied.

## Ineffectiveness of Other Treatments

Other coatings. - Several inorganic materials were deposited on the surfaces of the crystallites:  $Ca(OH)_2$ , CuCl, and $Ca(NO_3)_2$ . The sample containing $Ca(NO_3)_2$  showed a small amount of ductility at high loads (fig. 6). The other materials were not effective in imparting ductility to the compacted specimens. Other samples were coated (after pressing and cutting to size) by a submersion in oil or by spraying with Krylon. Neither of these treatments produced ductility.

Removal of material from the surface. - It has been reported that water-polished single crystals of sodium chloride exhibit more ductility than crystals which have not been water-polished. This is supposedly due to the removal of surface regions of sodium chloride which have a high concentration of dislocations. Polycrystalline sodium chloride was treated in a similar manner; before the powder was pressed it was washed with a nearly saturated solution of sodium chloride (to prevent the whole sample from going into solution). Other polishing treatments tried on the uncompacted powder were some using methyl alcohol and some using chloroform. None of the specimens prepared from any of the surface-treated crystallites showed ductility. It must be mentioned that, although the polishing may remove regions of high dislocation concentration from the uncompacted powder, the pressing process itself with the accompanying rupturing of grains would cause the formation of new regions of high dislocation concentration.

Compacted specimens were also surface-polished, one with alcohol and one with water. No ductility was found in the alcohol-polished specimen. The slight ductility for the water-polished specimen (fig. 6) is interpreted as being due to the penetration of the water along the grain boundaries; the water perhaps acts as a lubricant.

Grinding in helium. - The effect of various atmospheres upon the ductility of single crystals of sodium chloride is not known with

certainty. Results in the literature, however, seem to indicate that
air tends to decrease ductility (ref. 3). The results for specimens
whose powders were ground, screened, stored, and pressed in helium
showed no ductility.

Bulk treatments. - It was hoped that an annealing of compacted spec-
imens of sodium chloride might decrease the high dislocation regions of
the crystallites. Thus, a heat treatment was tried, but the resultant
specimens did not possess any ductility. Other annealed samples, the
crystallites of which were treated with various salt solutions prior to
pressing, were likewise not ductile.

Another type of bulk treatment used was X-irradiation. It has been
reported that such a treatment increases the length of the "foot" of the
load-deflection curve for single crystals (ref. 1). In one of the 20-
hour irradiated polycrystals, such a long foot was found (see fig. 7).
There was no general enhancement of ductility.

## EXPERIMENTAL DETAILS AND DISCUSSION FOR MAGNESIUM OXIDE

In general, specimens were fabricated and tested in the same manner
as was the sodium chloride. The magnesium oxide used was Fisher Scien-
tific analytical reagent grade with a particle size of about 0.7 micron.
After hydraulic pressing of the material and exposure to air for about
3 hours, the samples would exhibit delayed cracking without the applica-
tion of any external force; uncracked specimens could be cut from unfired
compacts if the compacts were stored out of contact with air for a mini-
mum of 3 weeks. The density of the specimens was never over 60 percent
of the theoretical value. The "green" strength was much greater than ex-
pected. Figure 8 gives the load-deflection curves of such specimens
after a heat treatment of 1000° F for 10 hours in vacuum; nearly straight-
line relations exist between load and deflection. The slopes of the
curves increase with increasing hydraulic pressure. None of the samples
tested exhibited the accepted Young's modulus, the experimental values
being much too low. Other unfired samples were prepared from 40-micron-
particle-size material; the "green" strength of such material was so poor
that the samples broke before specimens could be fabricated from them.
No further work was done with 40-micron material.

Of course, in order to obtain a material which had the properties
of a magnesium oxide ceramic, the compacts had to be fired. The curve
marked "untreated" in figure 9 gives the results for a specimen fired at
2250° F for 10 hours in an argon atmosphere. The entire load-deflection
curve is nonlinear and is concave upward; the slope near the fracture
point is very close to the slope predicted from the published modulus
($24.9 \times 10^{11}$ dynes/sq cm, ref. 6).

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The other curves in figure 9 are for samples prepared from powders which contained vapor-deposited metal. These powders were pressed at 50,000 pounds per square inch and fired under the same conditions as the untreated specimen. All the curves are nonlinear. The results in figure 9 are of a preliminary nature, and more experiments are needed before any conclusions may be drawn regarding the relative magnitude of the effects with various coatings. The chemical analyses of the materials before and after firing are listed in table II. The silver and magnesium were lost during firing, while the nickel remained. However, it is fairly certain that the nickel remaining after firing was altered in character, as indicated by the disappearance of the color due to the nickel.

CONCLUSIONS

It is possible to produce polycrystalline sodium chloride bodies which possess a small degree of ductility. For such materials the total strain can be three to four times the elastic strain. The ductility produced thus far in polycrystalline sodium chloride has always been accompanied by a decrease in tensile strength. The most effective method for producing this ductility to date is vapor deposition of a small amount of silver on each grain of material before compacting. The silver appears to act as a surface-active agent on the individual crystallites. Another method for producing ductility is coating each grain before compacting with a micron layer of stearic acid. The stearic acid perhaps acts merely as a lubricant.

Metallic deposits of the type which has been successful in producing ductility in polycrystalline sodium chloride have been used on samples of magnesium oxide before pressing; however, such metallic deposits are destroyed by the sintering process required to produce a ceramic body of the magnesium oxide.

Lewis Research Center
    National Aeronautics and Space Administration
        Cleveland, Ohio, June 25, 1959

APPENDIX - THE EQUATION FOR THE SLOPE

OF THE LOAD-DEFLECTION CURVES

The slope  S  of the Hooke's law load-deflection curve (fig. 3) was calculated from the following equation:

$$S = \frac{L}{D} = \frac{2}{3}\, Ybh\left(\frac{l}{2} - \frac{2}{3}\, c\right)$$

where  L  is the load, D  is the deflection of the loading points, Y  is Young's modulus, and  b, c, h, and  $l$  are defined in figure 1.  This formula is applicable to the four-point bending of beams with rectangular cross sections.

The slope equation can readily be derived from general equations in any textbook on the subject (ref. 8, p. 92, eq. (58), and p. 159, third eq.).

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TABLE 1. - SUMMARY OF TREATMENTS ON SODIUM CHLORIDE

| Type of treatment | Material | Average thickness of layer | Details of treatment | Results |
|---|---|---|---|---|
| Surface treatment of crystallites<br>1. Vapor deposit<br>(a) Metal<br>(1) Simple | Ag | 12 Å | NaCl ground under chloroform; chloroform evaporated in vacuum; Ag vapor-deposited | Ductile |
| | Ag | 2 to 7 Å | Vapor deposit on 325-mesh NaCl | |
| | Au | 5 Å | Vapor deposit on 325-mesh NaCl | Not ductile |
| | Cu | 19 Å | | |
| | Mg | 5 Å | | |
| | Cr | 0.5 Å | | |
| (2) Composite | Ag and Cu | 4 Å(Ag), 19 Å(Cu) | Ag deposited first, then Cu | |
| | Cu and Ag | 8 Å(Ag), 8 Å(Cu) | Cu deposited first, then Ag | |
| (b) Salt | AgCl | 8 Å | Evaporated from platinum boat | |
| 2. Liquid treatment<br>(a) Deposit | Stearic acid | 0.5 Micron | NaCl placed in chloroform solution of stearic acid; chloroform evaporated | Ductile |
| | Stearic acid | 0.2 to 0.5 Micron | Ether solution used; liquid centrifuged off | |
| | Ca(NO$_3$)$_2$ | 0.3 Micron | Acetone solution used; liquid centrifuged off | Slightly ductile |
| | CuCl | 0.3 Micron | Alcohol solution of CuCl$_2$ used; liquid centrifuged off; reduced to CuCl in 1/2 atm H$_2$ for 4 hr at 1000° F | Not ductile |
| | Ca(OH)$_2$ | ------ | NaCl powder placed in saturated NaCl solution of CaCl$_2$; liquid centrifuged off; powder then placed in saturated NaCl solution of NaOH; liquid centrifuged off | |
| (b) Polishing | ------ | ------ | Powder water-polished in saturated NaCl solution; liquid centrifuged off | |
| | ------ | ------ | Methyl alcohol polish; liquid centrifuged off | |
| | ------ | ------ | Chloroform polish; liquid evaporated | |
| 3. Gas adsorption | ------ | ------ | NaCl ground, screened, stored, and pressed in helium | |
| Surface treatment of specimen<br>1. Vapor deposit | Ag | ------ | Vapor deposition on tension and compression sides | Not ductile |
| 2. Liquid<br>(a) Deposit | Stearic acid | ------ | Specimen placed in ether solution of material several seconds and removed | |
| | Oil | ------ | Specimen placed in oil several seconds and removed | |
| | Krylon | ------ | Specimen sprayed with Krylon | |
| (b) Polishing | ------ | ------ | Specimen placed in water several seconds and removed | Slightly ductile but weak |
| | ------ | ------ | Specimen placed in alcohol several seconds and removed | Not ductile |
| Bulk treatment<br>1. Heat | ------ | ------ | Compacted specimen in vacuum at 1000° F for 15 hr | Not ductile |
| | Ca(OH)$_2$ | ------ | Pretreatment described for Ca(OH)$_2$ above; compacted specimen in vacuum at 1000° F for 15 hr | |
| 2. X-irradiation | ------ | ------ | X-irradiated 5 and 20 hr in helium; X-ray unit at 50 kv and 50 ma; sample 6 in. from target | Not ductile (one of two 20-hr samples had large "foot" on load-deflection curve) |

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TABLE II. - ANALYSES OF METALLIC

DEPOSITS ON MAGNESIUM OXIDE

| | Sample containing nickel, percent nickel | Sample containing metallic magnesium, percent metallic magnesium | Sample containing silver, percent silver |
|---|---|---|---|
| Before firing | [a]0.058 | 0.7 | 0.070 |
| After firing | [a].075 | <.01 | <.002 |

[a]These values are the same within experimental error.

Figure 1. - Bending jig.

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Bell jar

Vacuum chamber

Hammer

W-wire

Copper leads

Fins

Motor

Container for sodium chloride

CD-6668

To vacuum pump
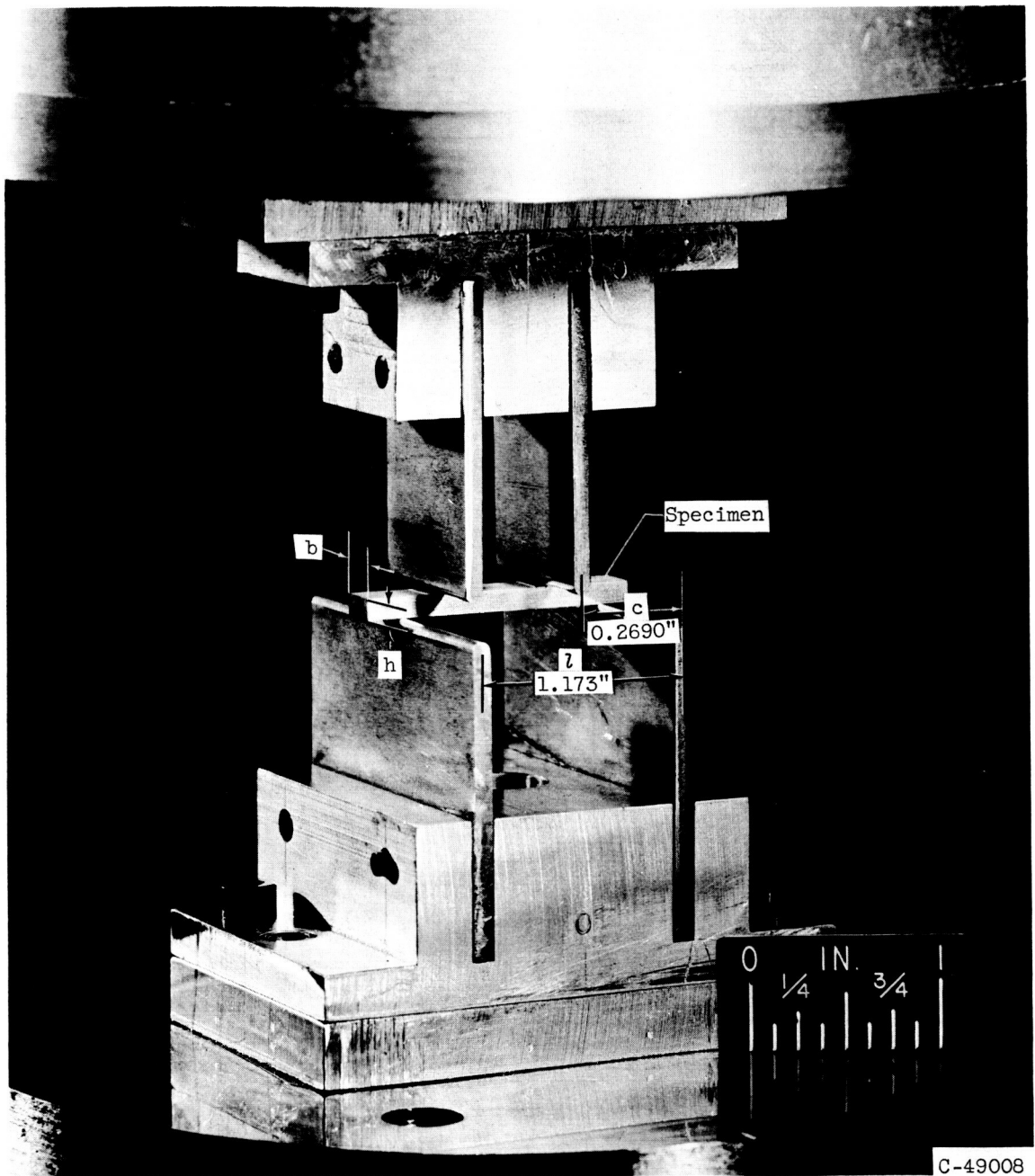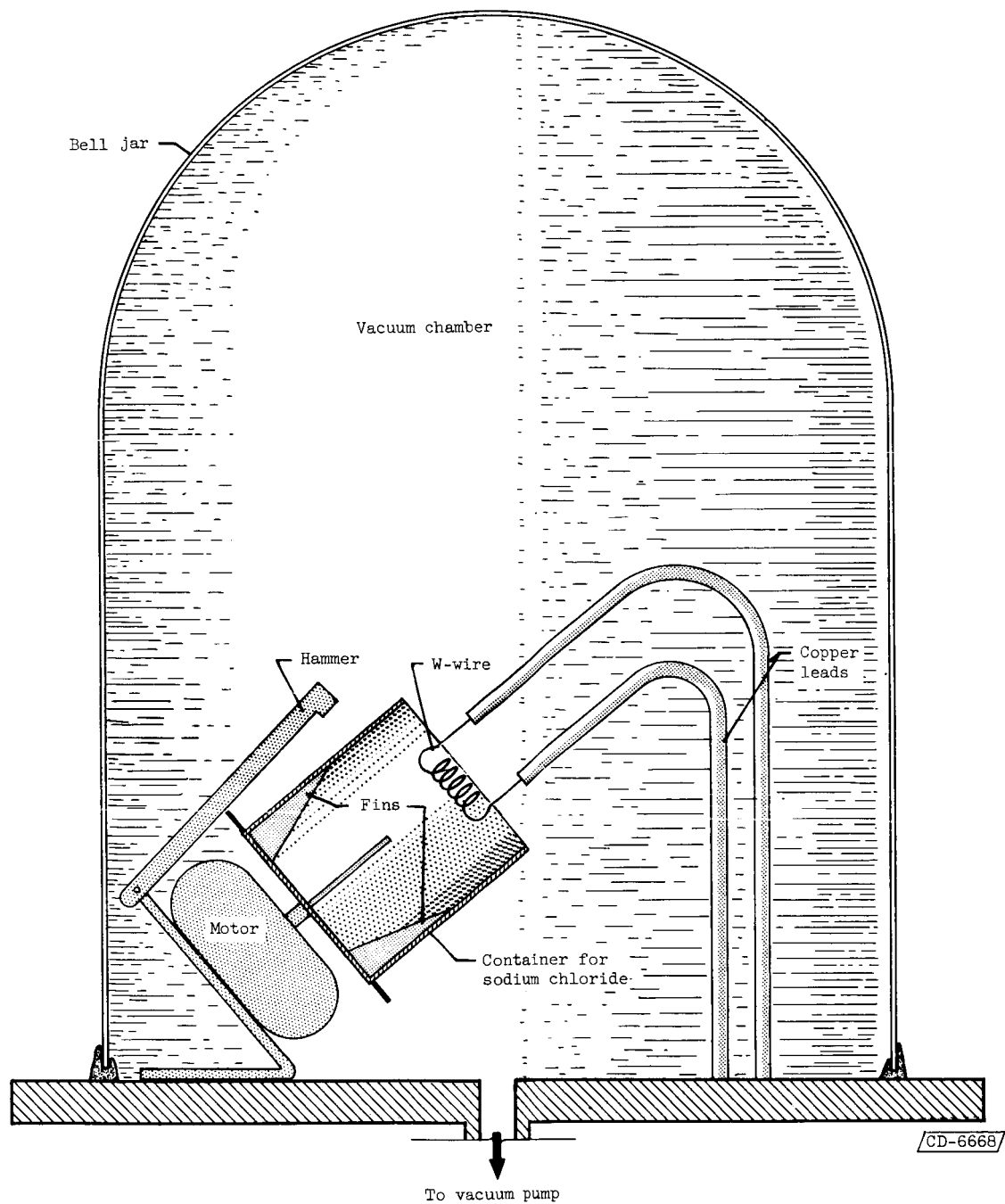
Figure 2. - Vapor-plating apparatus.
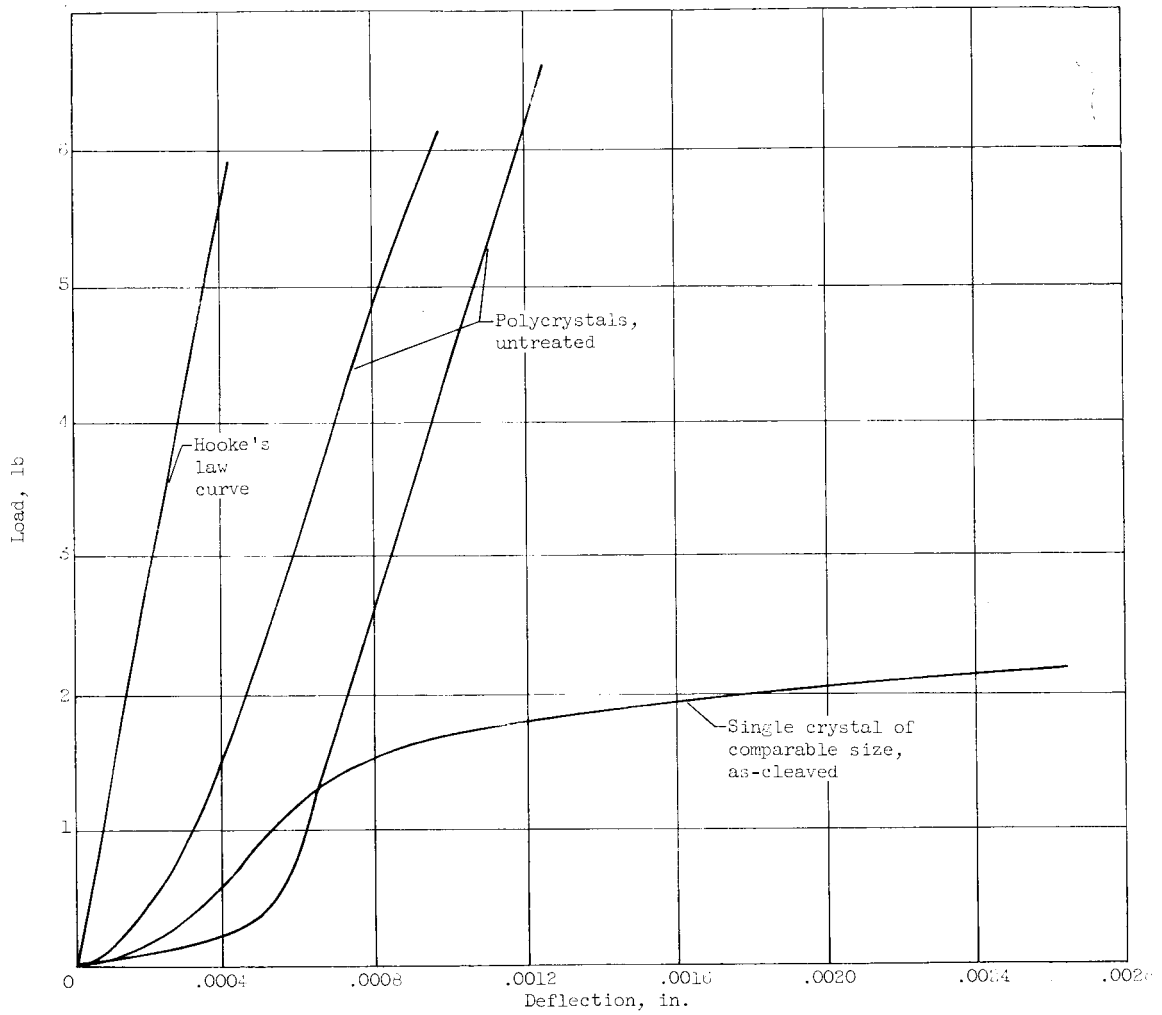
Figure 3. - Typical load-deflection curves for bending of polycrystalline and single-crystal sodium chloride.

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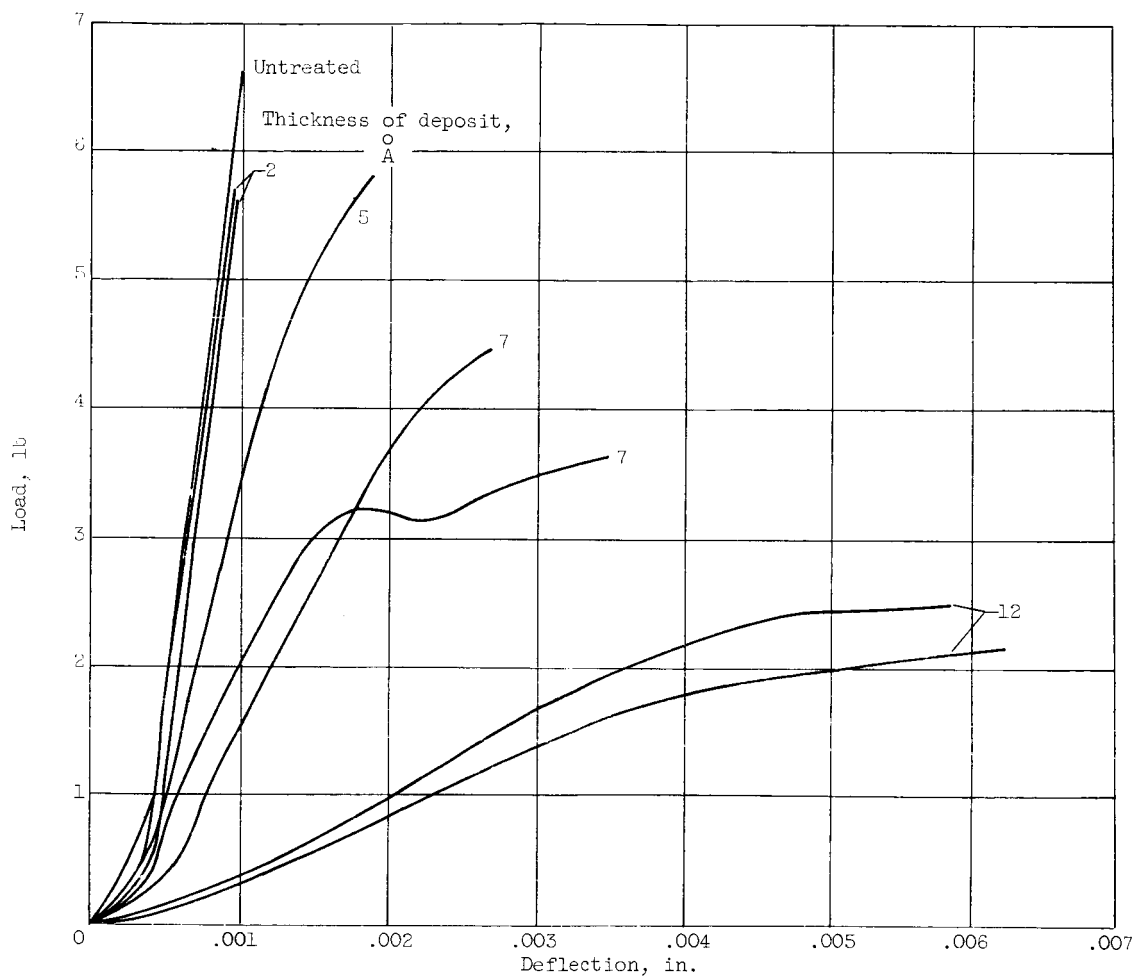


Figure 4. - Effect of silver on ductility of sodium chloride.
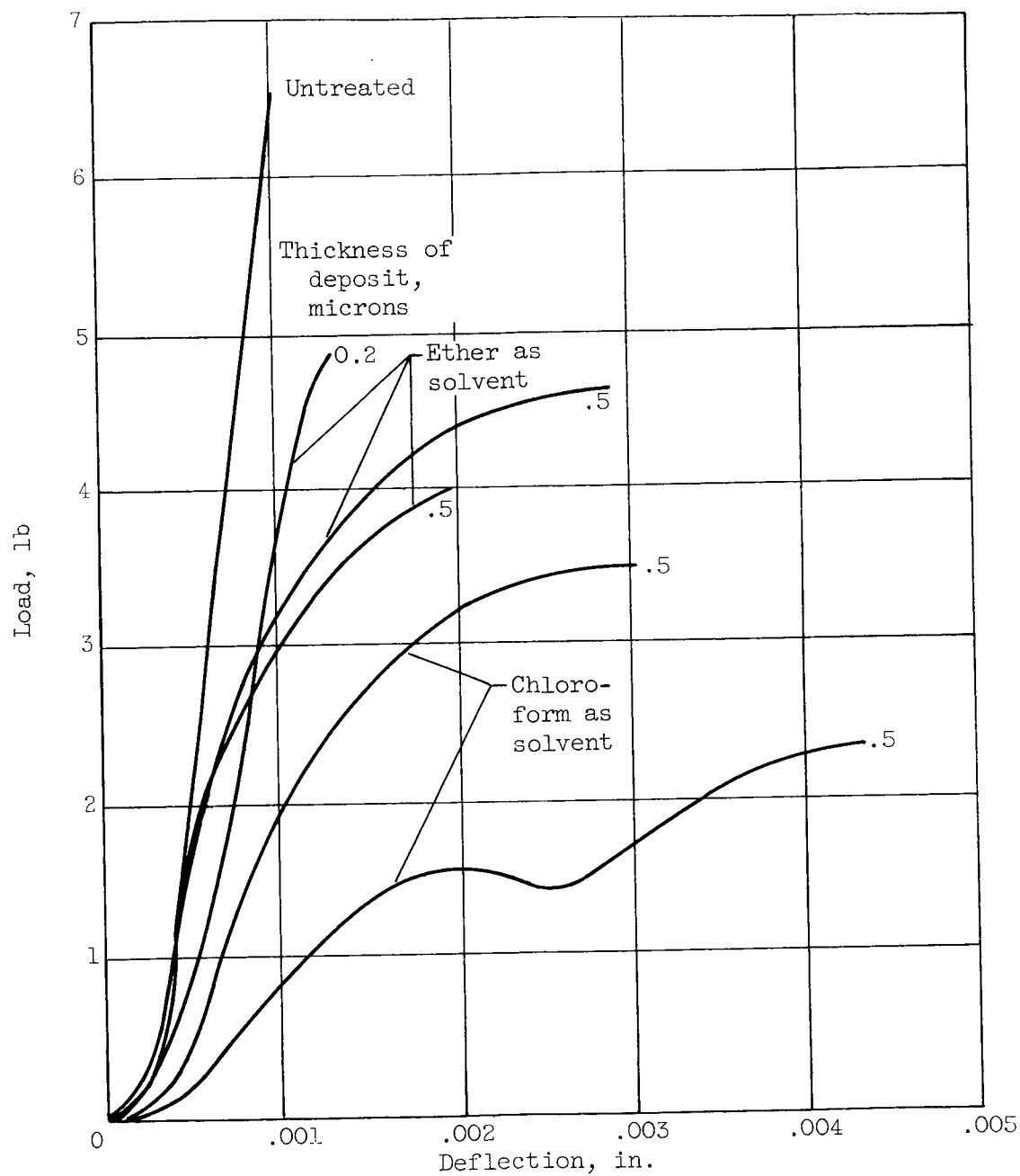
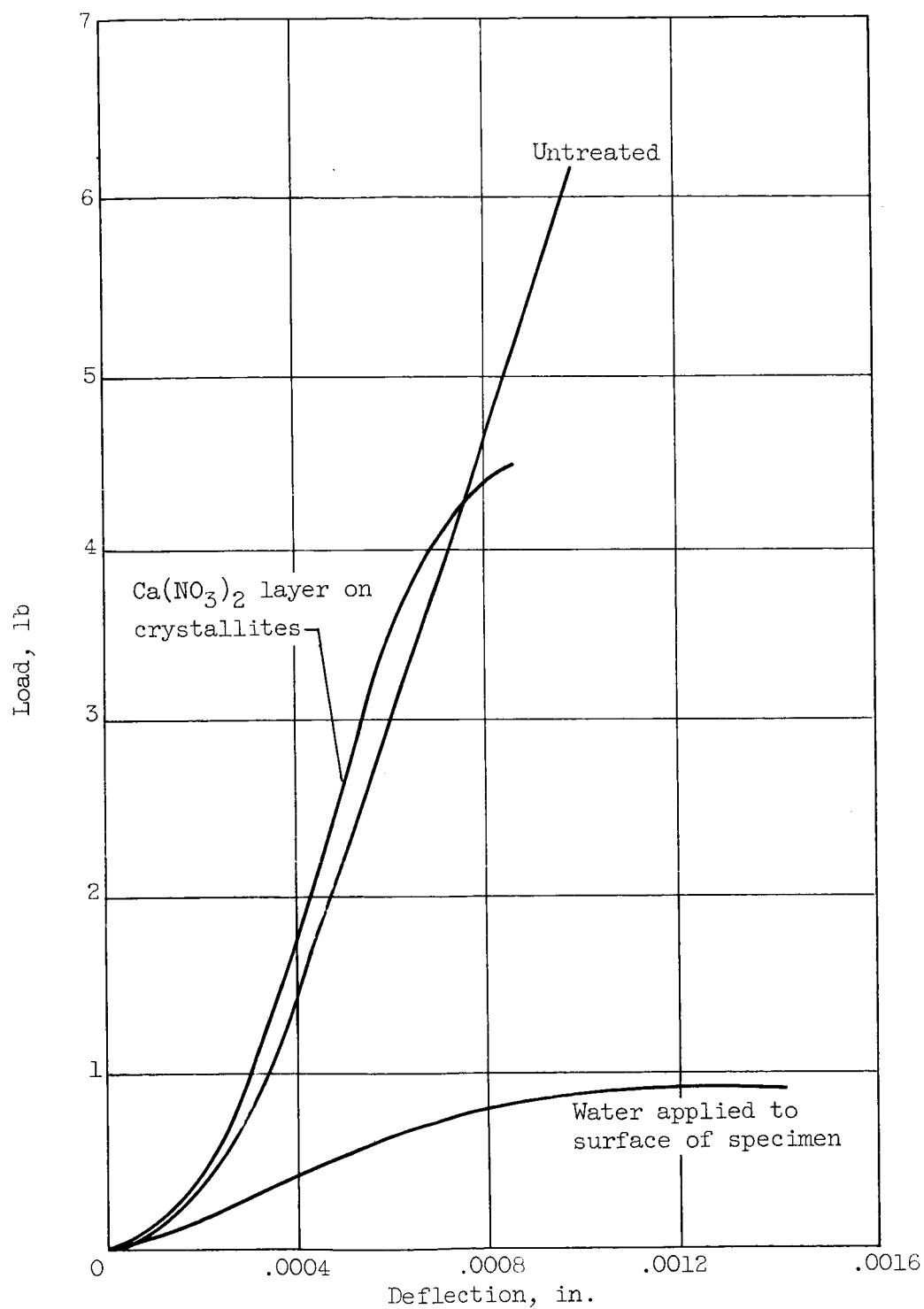Figure 5. - Effect of stearic acid on ductility of sodium chloride.

Figure 6. - Effect of $Ca(NO_3)_2$ and water treatments on ductility of sodium chloride.

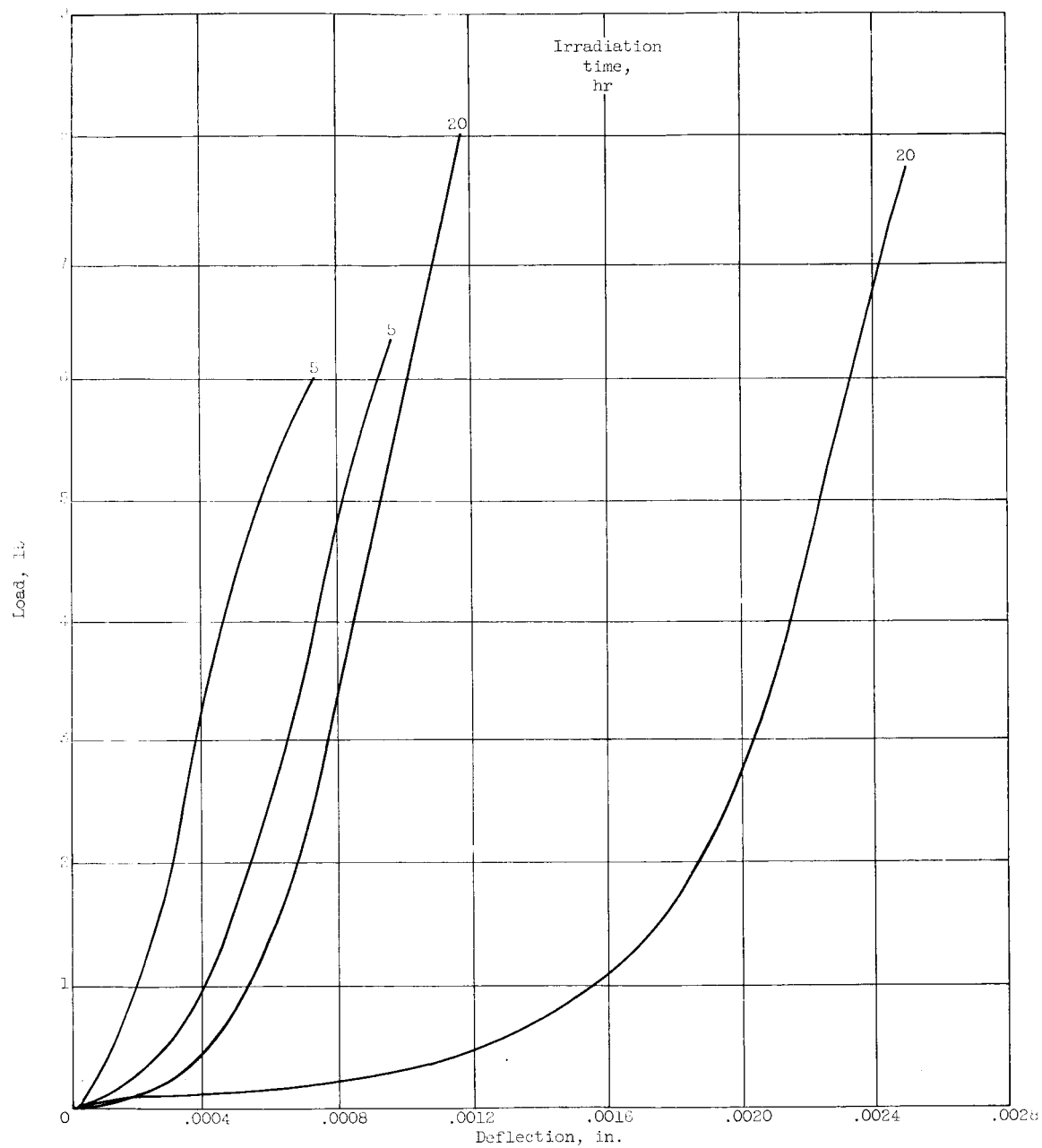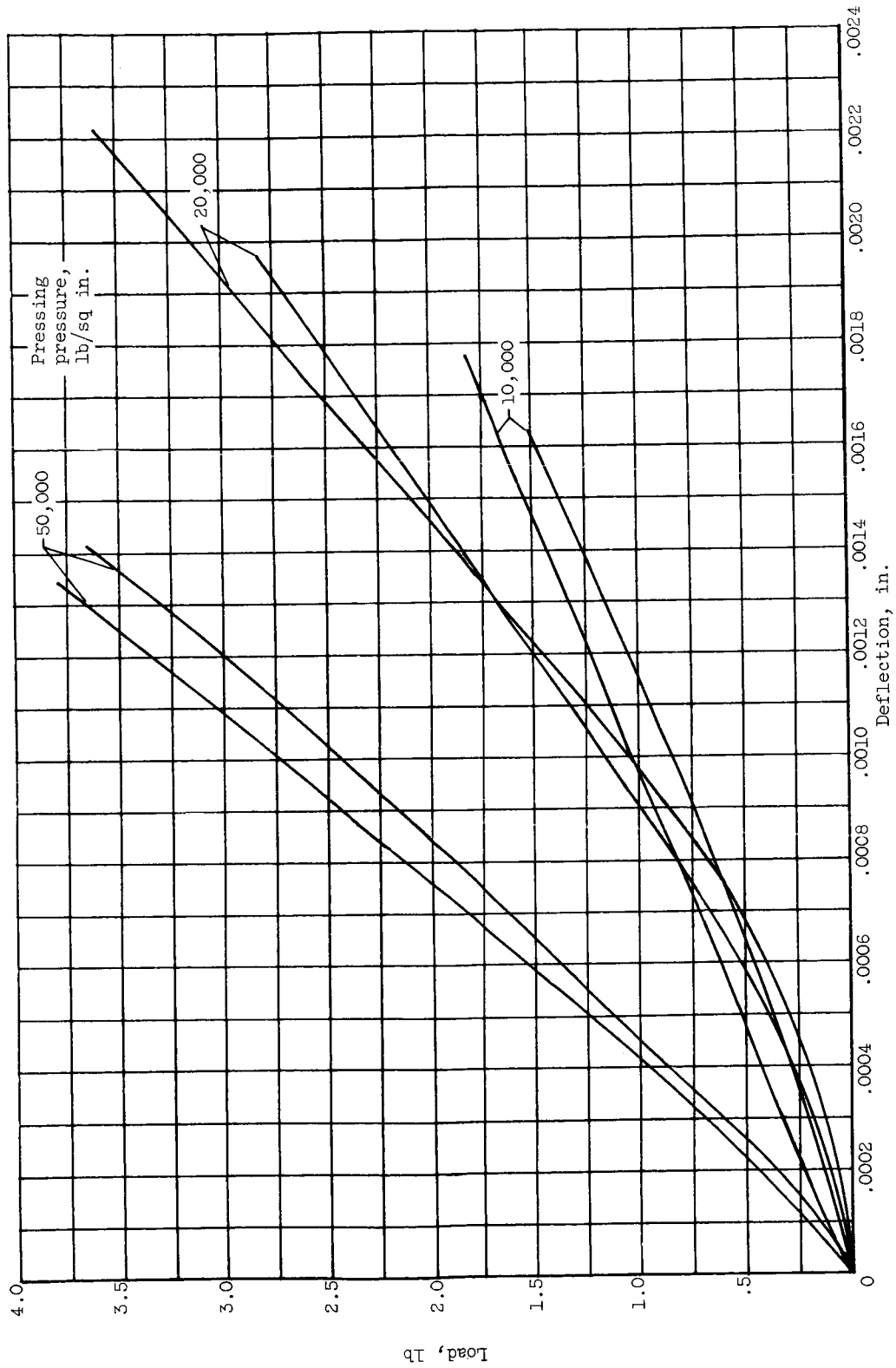Figure 7. - Effect of X-irradiation on polycrystalline sodium chloride.

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Figure 8. - Effect of pressing pressure on load-deflection curves of unfired magnesium oxide. Average specimen size, 0.125 by 0.25 by 1.6 inches.
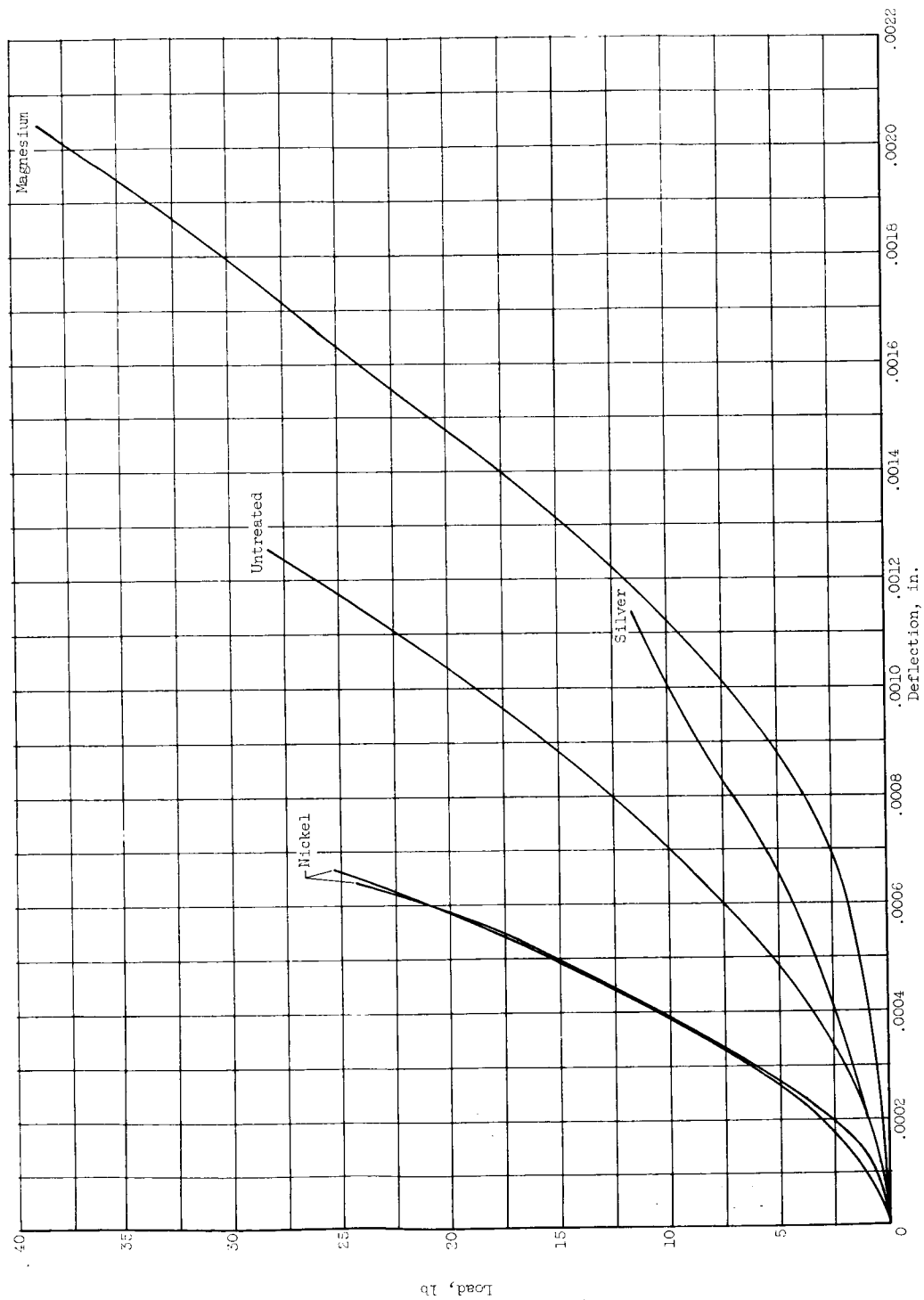
Figure 3. - Effect of various vapor deposits on load-deflection curves of sintered magnesium oxide. Average specimen size, 0.11 by 0.21 by 1.6 inches.